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## EFFECT OF HEAT TREATMENT ON STIFFNESS AND DAMPING OF SiC/Ti-15-3

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### Abstract

The effect of heat treatment on material properties of SiC/Ti-15-3 was measured by vibration tests. Heat treatment changes the microstructure, which was found to stiffen the matrix and reduce its damping capacity. Test results illustrate how these changes in the matrix affect the corresponding properties of the composite. Measurements show that heat treatment affects damping properties of the composites to a greater extent than stiffness properties. The extent of the changes in mechanical properties is shown to depend on heat treatment temperature and exposure time.

### Introduction

Mechanical loading and thermal aging introduce damage into metal matrix composites that can eventually lead to the failure of composite structures. If the initiation and development of microscale damage could be tracked nondestructively, the composite structure could then be repaired or replaced prior to catastrophic failure. Toward this end, noninvasive inspection techniques have been developed to detect damage-induced changes in the microstructure of fiber reinforced composite materials.<sup>1-4</sup> An alternative to these traditional *local* NDE techniques is to measure changes in *global* structural dynamics that result from changes in the material properties.<sup>5-10</sup>

In Ref. 10, a sensitive technique was developed to detect small changes in material properties of metal matrix composites, such as those caused by damage due to mechanical loading and/or thermal aging. Free vibration response to a low-amplitude impact load was monitored, and the measured vibration frequencies were used to determine the flexural stiffness of the test specimens. Recent work in acousto-ultrasonics has showed that the propagation of elastic stress waves is attenuated by damage in the material.<sup>5-8</sup> This attenuation may cause a significant change in the damping properties of the material. In the present work, the vibration monitoring approach in Ref. 10 is used to determine how damping properties are affected by heat treatment. The results indicate that modal frequency and damping properties can be used to nondestructively detect the microstructural changes in a SiC/Ti-15-3 composite caused by heat treatment.

### Methods

The material was fabricated using a proprietary process in which alternating layers of SCS-6 SiC fibers were consolidated between foils of Ti-15V-3Cr-2Sn-3Al (Ti-15-3). Eight rows of fibers were used, resulting in a total composite thickness of approximately 0.08 in. The fiber volume fraction was nominally 34 percent. Microstructural details of the material can be found in Refs. 11 and 12. An unreinforced Ti-15-3 plate was fabricated by laminating foils in a process similar to that used in making the composite. In this way, the mechanical properties of the unreinforced plate would be nearly the same as those of the matrix within the composite.

Five unidirectionally reinforced  $[0^\circ]_8$  and two monolithic Ti-15-3 (matrix only) specimens were tested. The specimens were originally designed for thermomechanical fatigue testing, and were therefore of nominal dimension 15.2 by 1.27 by 0.167 mm (6 by 0.5 by 0.08 in.), with a reduced gage section, as shown in Fig. 1. Individual specimens were cut from composite panels and the Ti-15-3 plate using an EDM process.

### Heat Treatment

Each test specimen was subjected, in a vacuum, to one of three heat treatments to vary the microstructure and the resulting mechanical properties of the matrix:

- (1) 700 °C/24 hr + f.c.
- (2) 700 °C/24 hr + f.c. + 427 °C/24 hr + f.c.
- (3) 788 °C/15 min + W.Q. + 300 °C/24 hr + f.c.

where f.c. is furnace cooled and W.Q. is water quenched.

The microstructures differ primarily in the size, shape, and location of the alpha-titanium phase.<sup>11</sup> The microstructures of the unreinforced Ti-15-3 are shown in Fig. 2. The corresponding mechanical properties of the titanium as measured in tensile tests<sup>13</sup> are given in Table 1. The high strength and stiffness produced by heat treatments 2 and 3 are a result of the high volume fraction of fine alpha-titanium precipitates.

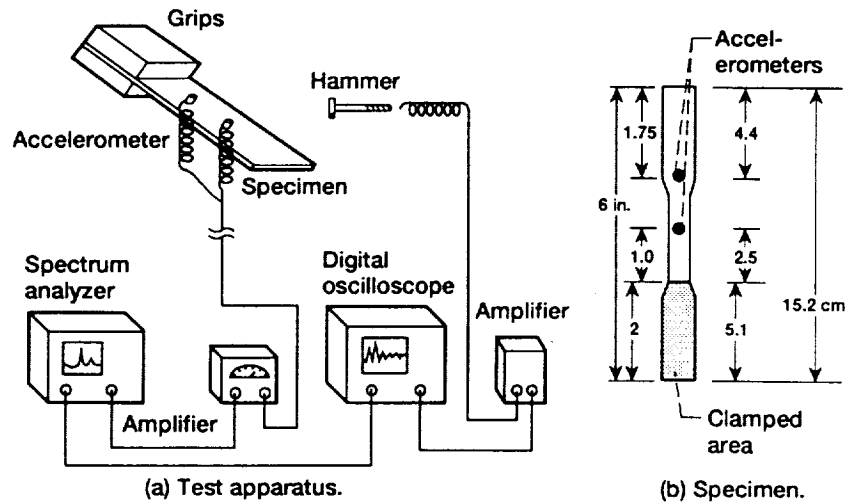


Figure 1.—Test specimen and vibration measurement apparatus.



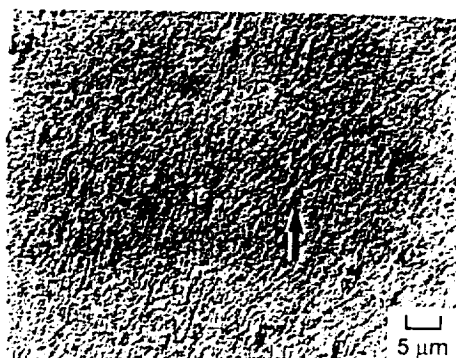
(a) As-received Ti-15-3.



(b) After heat treatment 1.



(c) After heat treatment 2.



(d) After heat treatment 3.

Figure 2.—Effect of heat treatment on microstructure of Ti-15V-3Cr-3Sn-3Al. Note the size, shape and distribution of the α-Ti precipitates (indicated by arrows) at the grain boundaries and in the grain interiors.

TABLE 1.—TENSILE PROPERTIES OF  
HEAT TREATED Ti-15-3

(Ref. 13)

Heat treatment number	E		$\sigma_{uts}$		$\epsilon_f$ percent (inelastic)
	GPa	Msi	MPa	ksi	
None	86.9	12.6	931	135	12
1 700 °C/24 hr	82.7	12.0	869	126	21
2 700 °C/24 hr + 427 °C/24 hr	107	15.5	1413	205	7
3 788 °C/15 min + 300 °C/24 hr	103	15.0	1448	210	.03

The very fine alpha particles resulting from heat treatment 3 have been shown to localize slip, reducing the ductility to negligible levels.<sup>14</sup> The microstructure of the in-situ matrix material in the SiC/Ti-15-3 composites is nominally the same as that shown here for the monolithic matrix.<sup>11</sup>

#### Vibration Testing

The test specimens were cantilevered in a clamping fixture as shown in Fig. 1. Vibration was induced by applying an impact load at the free end of the specimens with an instrumented hammer. Two lightweight (140 mg) piezoelectric accelerometers were mounted on the specimens with cyanoacrylate glue at the positions shown in the figure. The impact force and acceleration time histories were digitally recorded at a rate of 1 MHz for 8.2 msec after the initial impact. Each specimen was tested in the initial, as-received condition, and after exposure to one of the three heat treatments.

#### Frequency Response Function

The frequency response,  $H(\omega)$ , of each test specimen was calculated from the force and acceleration time histories:<sup>15</sup>

$$H(\omega) = \frac{A(\omega)}{F(\omega)} \quad (1)$$

where  $H(\omega)$  and  $A(\omega)$  are the force and acceleration response histories expressed in the frequency domain, which are defined from the measured time histories using the Fourier transform;

$$F(\omega) = \int_{-\infty}^{\infty} f(t)e^{-i\omega t} dt \quad (2)$$

where  $F(\omega)$  denotes the force history expressed in the frequency domain and  $f(t)$  is the measured force data, in the time domain. The frequency domain results were calculated from the time histories using a Fast Fourier Transform (FFT) algorithm.<sup>16</sup>

To minimize the effects of signal noise on the measurements, an averaging process was used to calculate the frequency response functions. The results shown here represent the averaged values of 10 frequency response functions calculated from 10 separate vibration tests on each specimen. This approach was shown to determine resonant frequencies of the test specimens with a measurement variability of  $\pm 0.2$  percent.<sup>10</sup>

Changes in flexural stiffness due to heat treatment were determined from the measured changes in resonant frequencies. If it is assumed that the vibration of the test specimens can be described by a simple Euler beam model,<sup>17</sup> the resonant frequencies  $\omega_n$  are given by

$$\omega_n = \frac{(\beta_n L)^2}{2\pi L^2} \sqrt{\frac{EI}{\rho A}} \quad (3)$$

where  $n$  designates the vibration mode,  $L$  is the beam length,  $\rho A$  is mass density per unit length,  $EI$  is the flexural stiffness<sup>18</sup> of the composite and  $\beta_n$  is a constant of integration, determined by the beam boundary conditions.

High frequency vibration modes are generally more sensitive to material damage than are the lower frequency modes.<sup>10</sup> Therefore, modal properties (frequency and damping) for the 26 kHz bending mode were measured for the unidirectional composites, and the same measurements were taken for the 14 kHz mode of the monolithic matrix specimens. In each case, these correspond to the fifth or sixth flexural vibration mode for the specimen.

#### Damping Measurement

Damping is the dissipation of kinetic energy by a structure. Test data have shown that microstructural changes in the material may affect structural damping properties as well as the resonant frequencies.

To obtain some measure of the damping from the free vibration response, a single vibration mode was examined by bandpass filtering the acceleration history such that the response at all but one resonant frequency was filtered out. Recently, a linearized logarithmic decrement technique was used<sup>19,20</sup> to obtain a measure of the structural damping in graphite/aluminum composites from the free vibration response. This approach was followed here, and is outlined briefly below.

If the structural damping is of the viscous type, the dissipative force varies with the velocity of the motion, which causes the vibration to decay exponentially with time:<sup>21</sup>

$$x(t) = X_0 e^{-\xi \omega t} \cos(\omega_d t - \phi) \quad (4)$$

where  $x(t)$  is the displacement as a function of time,  $\xi$  is the viscous damping coefficient,  $\omega$  is the undamped vibration frequency,  $\phi$  is the phase angle and  $X_0$  is the initial amplitude of the motion. The damped vibration frequency,  $\omega_d$  is given by

$$\omega_d = \omega \sqrt{1 - \xi^2} \quad (5)$$

Equation (4) represents a sinusoidal motion within an exponentially decaying envelope. The decay rate is characterized by the damping coefficient,  $\xi$ :

$$\ln \frac{X_0}{x(t)} = \xi \omega t \quad (6)$$

In general, structural damping coefficients are small, that is,

$$\xi \ll 1 \quad (7)$$

Therefore, the acceleration measured at a point on the test specimen is given by

$$\ddot{x} = -\omega_d^2 x \quad (8)$$

where  $\omega_d = \omega$  from Eqs. (6) and (7). The damping coefficient can therefore be obtained from the acceleration data. From Eqs. (6) and (8).

$$\xi \omega t = \ln \frac{\ddot{x}_0}{\ddot{x}(t)} \quad (9)$$

where  $\ddot{x}$  is the decaying amplitude of the measured acceleration, and  $\ddot{x}_0$  is the maximum (initial) acceleration. The largest damping coefficient measured for the material used in this work was approximately  $3 \times 10^{-5}$ , which confirms the validity of the assumption stated in Eq. (7).

### Results and Discussion

Two monolithic Ti-15-3 matrix specimens and five unidirectional  $[0^\circ]_8$  composite specimens were vibration tested, as discussed in the previous section, before and after being exposed to one of the three heat treatments shown in Table 1. Changes in modal stiffness and damping were calculated from those measurements. Test data for representative test specimens are given in Figs. 3 to 8. The effects of the three heat treatments on vibration properties are compared in Figs. 9 and 10, and those results are tabulated in Tables 2 and 3.

#### Vibration Measurements

A typical impact force applied to composite specimen number 52 with the instrumented hammer had a duration of approximately 50  $\mu$ sec, and is shown in Fig. 3. The impact force was recorded for approxi-

mately 8 msec. The first millisecond of one typical record is plotted in the figure, to show details of the lower amplitude multiple impacts that sometimes occurred within 1 msec of the initial pulse. The corresponding frequency spectrum, obtained from the force history using an FFT algorithm,<sup>16</sup> shows that the impact force has significant frequency components up to approximately 35 kHz. The resulting measured acceleration response for specimen number 52 is shown in Fig. 4.

Figure 5 shows the corresponding frequency response functions calculated for specimen number 52. The dashed line is calculated from the force and acceleration measurements in Figs. 3 and 4, which were taken before the specimen was exposed to heat treatment. The solid line was measured in a similar manner after subjecting the composite specimen to heat treatment number 1. The upward shift in resonant frequencies after heat treatment is the result of an increase in stiffness of the material. In the figure, the 26 kHz mode increases in frequency by approximately 3 percent. Using Eq. (3), this corresponds to a 6.1 percent increase in bending stiffness of the composite.

In addition to the increase in stiffness, heat treatment decreases the bandwidth of the frequency response peaks, as shown in Fig. 5. Frequency response bandwidth is a measure of the structural damping.<sup>21</sup> This suggests, therefore, that the damping in the material is significantly reduced by heat treatment. The signal obtained by bandpass-filtering the acceleration data for the 2 kHz bending mode of specimen number 52 is shown in Fig. 6. To isolate the 26 kHz mode, all frequencies below 23 kHz and above 30 kHz were eliminated in the filtering process. The filtered signal decays by a logarithmic decrement, and the damping coefficient is determined from the averaged slope of the data, as shown in Fig. 7. The effects of the three heat treatments on damping properties are shown in Table 3.

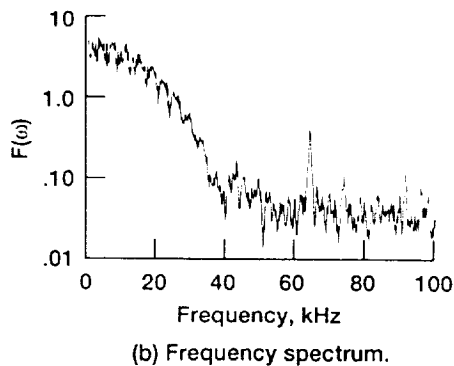
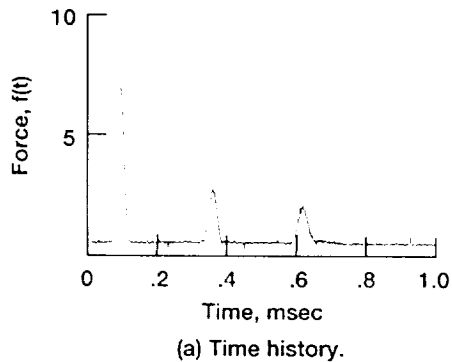


Figure 3.—Time history and frequency spectrum of a typical impact force applied to as-received unidirectional composite specimen #42 with the instrumented hammer.

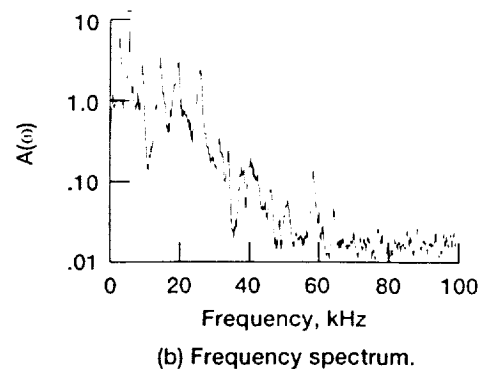
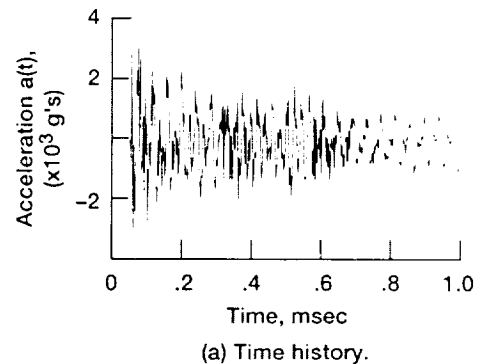


Figure 4.—Time history and frequency spectrum of a typical acceleration response of as-received unidirectional composite specimen #52 subjected to the impact force shown in Figure 3.

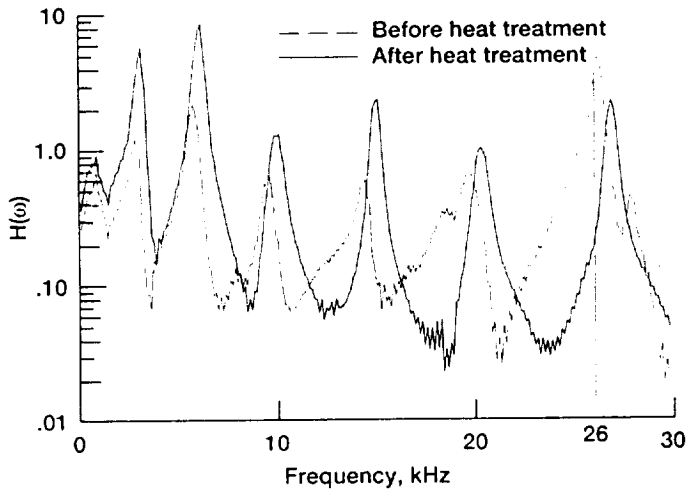


Figure 5.—Effect of heat treatment #1 on frequency response of unidirectional composite specimen #52. Dashed line is calculated from data in Figures 3 and 4. Frequency and damping measurements were taken for the 26 kHz mode.

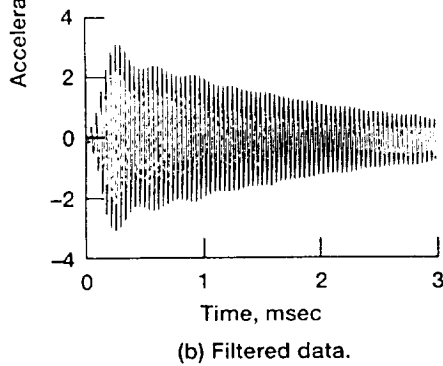
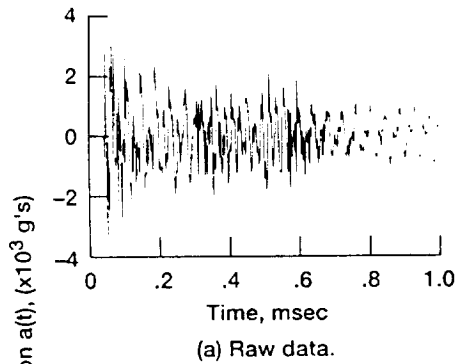


Figure 6.—Effect of 23-30 kHz bandpass filtering on acceleration response history from Figure 4.

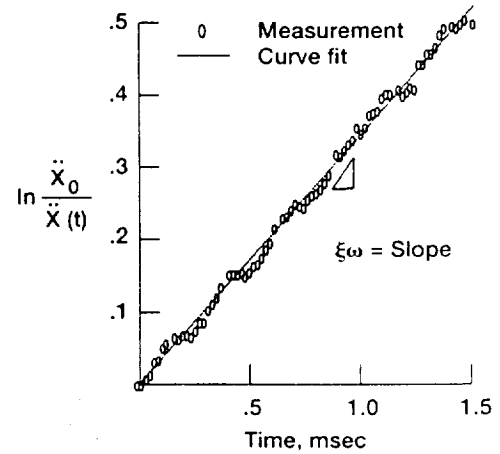


Figure 7.—Logarithmic decrement decay rate measured from filtered signal in Figure 6. The modal damping coefficient for the material is determined from the slope of the least-squares curve fit line.

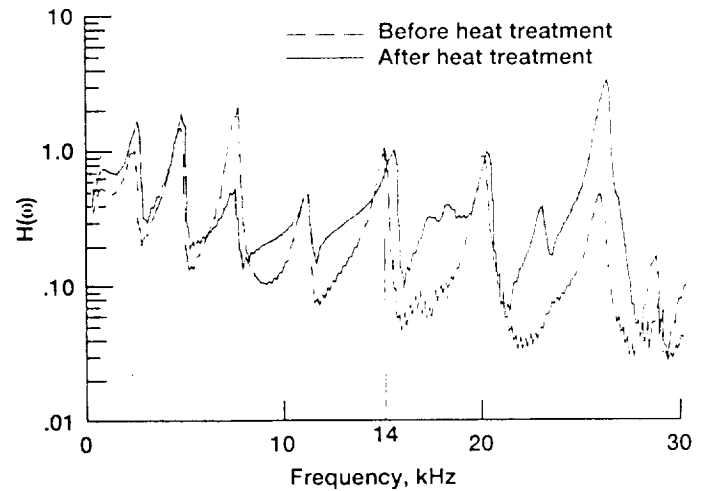


Figure 8.—Effect of heat treatment #1 on frequency response of monolithic Ti-15-3 matrix specimen #M1. Frequency and damping measurements were taken for the 14 kHz mode.

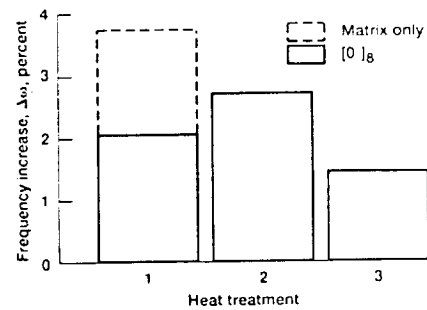


Figure 9.—Effect of heat treatment on resonant frequencies of Ti-15-3 matrix specimens (14 kHz mode) and on unidirectional SiC/Ti-15-3 composites (26 kHz mode).

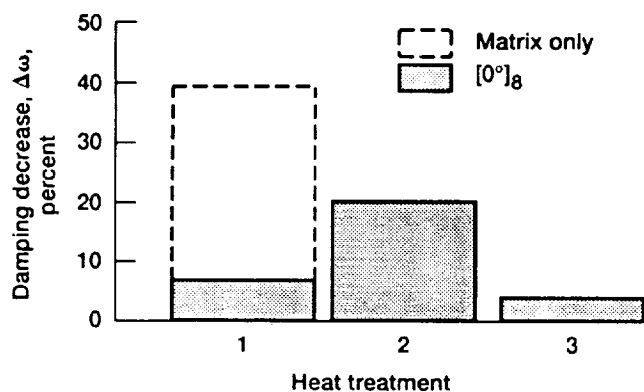


Figure 10.—Effect of heat treatment on damping coefficients of Ti-15-3 matrix specimens (14 kHz mode) and on unidirectional SiC/Ti-15-3 composites (26 kHz mode).

TABLE 2.—FREQUENCY CHANGES DUE TO HEAT TREATMENT

Specimen <sup>a</sup>	Initial $\omega$ , kHz	After heat treatment number			Percent change
		1	2	3	
M1	14.52	15.13	-----	-----	+4.2
M2	14.52	15.01	-----	-----	+3.4
52	25.74	26.35	-----	-----	+2.4
53	25.74	26.23	-----	-----	+1.9
50	25.99	-----	26.72	-----	+2.8
51	26.23	-----	26.96	-----	+2.8
54	25.74	-----	-----	26.23	+1.9

<sup>a</sup>M1 and M2 are monolithic matrix specimens 50 to 54 are  $[0^\circ]_8$  composites.

TABLE 3.—DAMPING CHANGES DUE TO HEAT TREATMENT

Specimen	Initial <sup>a</sup> damping, $\xi$	After heat treatment number			Percent change
		1	2	3	
M1	1.90	1.32	-----	-----	-31
M2	1.90	.99	-----	-----	-48
52	2.06	1.89	-----	-----	-8.1
53	1.58	1.49	-----	-----	-5.6
50	2.86	-----	2.27	-----	-20.9
51	-----	-----	1.53	-----	-----
54	1.44	-----	-----	1.38	-4.1

## Matrix Properties

**Modulus.**—The effect of heat treatment on the frequency response function for monolithic matrix specimen number M1 is shown in Fig. 8. The dashed line is calculated from measurements taken in the initial, as-received condition, and the solid line is calculated from measurements taken after subjecting the composite specimen to heat treatment number 1. The shift in frequency response indicates that an increase in stiffness due to heat treatment also occurs in the monolithic matrix material.

The heat treatment resulted in an average 3.8 percent increase in frequency for the matrix specimens, as indicated in Table 2 and Fig. 9. Using the frequency-stiffness relationship in Eq. (3), this corresponds to a 7.7 percent increase in the flexural modulus of the titanium. This is slightly greater than the 6.1 percent increase in stiffness measured for the composite, after the same heat treatment.

In comparison, the data from Ref. 13 in Table 1 indicate a small decrease in tensile modulus after heat treatment number 1. This is surprising, since the microstructural changes that resulted from both of the other heat treatments caused substantial increases in modulus, as measured in the tensile tests. The measurement uncertainty for the data reported in Ref. 13 is approximately  $\pm 3.5$  GPa ( $\pm 0.5$  Msi), so a modulus change of the magnitude reported for heat treatment number 1 in Table 1 should be considered negligibly small.

**Damping.**—The damping coefficient for the 14 kHz vibration mode of the monolithic Ti-15-3 matrix specimens was measured, using the bandpass filtering technique discussed earlier. Measurements were taken before and after the matrix specimens were exposed to heat treatment number 1. The results, given in Table 3, show that heat treatment caused an average 40 percent decrease in damping for the matrix specimens.

## Composite Properties

**Modulus.**—Assuming that heat treatment affects only the matrix properties, the composite stiffness should be less sensitive than the monolithic matrix to heat treatment because the composite is only 66 percent matrix, and because most of the stiffness for the  $[0^\circ]_8$  composite is provided by the fibers.

Using the results discussed above for the monolithic matrix material, it can be assumed that heat treatment number 1 causes the in-situ matrix in the composite to increase in modulus by 7.7 percent. With a fiber modulus of 430 GPa (62 Msi)<sup>10</sup> and a modulus for the as-received matrix of 87 GPa (12.6 Msi) given in Table 1, a modulus change of 2 percent is expected for the composite, using a rule-of-mixtures calculation<sup>22</sup> based on a 34 percent fiber volume fraction for the composite.

Test measurements, shown in Table 2 and Fig. 9, indicate that the composite specimens increased in frequency by an average of 2 percent (a 4 percent increase in flexural modulus, using Eq. (3)) due to heat treatment. This is approximately twice the expected stiffness increase, based on the analysis above. Assuming that fiber stiffness is unaffected by heat treatment, this result suggests that:

(1) The in-situ matrix reacts differently to the thermal load than does the monolithic matrix; and/or

(2) Heat treatment increases the interfacial bond strength between fiber and matrix, transferring load more effectively to the fibers.

Examination of SEM photographs of the fiber/matrix interface region has indicated that the size of the reaction zone at the interface does not change noticeably due to these heat treatments. Fiber push-out tests are currently being conducted to determine if the interfacial bond strength



was affected by heat treatment. Indeed, recent work<sup>23</sup> has shown that a 500 °C/50-hr heat treatment increased the as-received interfacial bond strength by 32 percent for SiC/Ti-15-3 composites.

Comparison of the results shown in Fig. 9 for heat treatment number 1 with those for heat treatments number 2 and number 3 suggest that the increase in stiffness depends on the temperature and duration of heat treatment. Heat treatment number 2 was 24 hr longer than heat treatment number 1 (see Table 1), and therefore resulted in an additional stiffness increase, as shown in Fig. 9. Heat treatment number 3 had the same duration as number 1, but a lower temperature, and therefore resulted in a correspondingly smaller increase in stiffness.

**Damping.**—Heat treatment number 1 reduced the damping of the composite by only 7 percent, compared to an average decrease of 40 percent for the matrix alone, as shown in Fig. 10. This is because damping in the composite can be caused by several different dissipative mechanisms including frictional sliding between fiber and matrix, hysteretic damping in the fibers, and by hysteresis in the matrix. Damping in the monolithic matrix specimens can only be caused by the latter of these.

If matrix hysteresis is the primary source of damping in the composite, the damping sensitivity of the unidirectional composite to heat treatment should vary in proportion to the matrix content in the composite. Based on the 40 percent decrease in damping for the monolithic matrix specimens, we would therefore expect heat treatment number 1 to cause a 26 percent decrease ( $0.66 \times 40$ ) in damping for the unidirectional composite. In fact, the damping in the composites decreases by only 7 percent due to heat treatment number 1. This suggests that matrix hysteresis does not contribute significantly to flexural damping in the unidirectional composites, and that the damping properties of the fiber itself and the frictional sliding between fiber and matrix may be the primary sources of damping in unidirectional SiC/Ti-15-3 composites.

### Conclusions

Analysis of instrumented vibration tests indicate that heat treatment of SiC/Ti-15-3 changes the matrix microstructure, which stiffens the composite and reduces its damping capacity. Damping coefficients decreased between 4 to 20 percent in the unidirectional composites due to heat treatment, and resonant frequencies increased 2 to 3 percent. The mechanical properties of the monolithic matrix material are more sensitive to heat treatment than are those of the composite. The extent of the measured changes in properties is believed to depend on heat treatment temperature and exposure time. Test results indicate that matrix hysteresis is not the primary source of flexural damping in the unidirectional composites. Test results provide supporting evidence that heat treatment can increase fiber/matrix interfacial bond strength.

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